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The major effort related to facilities has been the upgrading of the multi-source electron beam deposition system which is used to prepare the ternary and pseudoternary compounds that are being studied under the program.

The vacuum chamber has been equipped with a Williamson noncontacting temperature sensing system for monitoring substrate temperature. With this system the temperature of the substrate is determined by computing the ratio of radiant energies emitted from the surface in two adjacent wavebands.

We have not purchased a rate monitor which uses Electron Impact Emission Spectroscopy (EIES). The currently commercially available instrument, supplied by Inficon, has the capability for controlling only two sources, whereas in our applications there is a need for control of up to four. Keywords: Intermetallic compounds, Superconductivity.

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Research Progress and Forecast Report

Superconductivity of Thin Film Intermetallic Compounds

Grant No. AFOSR-84-0347

March 31, 1985

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TABLE OF CONTENTS

I. INTRODUCTION

The study of thin-film superconducting compounds has become an important frontier of solid state science and a part of the technical and scientific base of both large-scale and small-scale applications of superconductivity. Both of these classes of applications should have increasing impact on industrial and military technologies in areas related to energy, electronics and information processing as the twentieth century approaches. The program described here, which began 1 September 1984, is designed to take advantage of the control of chemical composition and morphology possible with thin film techniques such as sputtering and electron beam co-evaporation. Materials fabricated and studied include the Chevrel phase compounds and low carrier density systems.[2] These are of potential technological significance. In addition, fundamental problems such as the interplay between magnetic order and superconductivity, and the nature of the mechanism for superconductivity in the heavy fermion materials[3] will be investigated. An important aspect of the work is the correlation of microscopic superconducting parameters determined by tunneling with composition and structure. Macroscopic properties of the superconducting state such as the critical field, critical current and critical temperature will also be studied and correlated with microscopic parameters, composition and structure.

Although the efforts described here by and large involve fundamental scientific studies, the materials efforts involved in preparing samples needed to test theoretical models and answer scientific questions are intimately related to efforts needed for the development of the technology. The optimization of the superconducting properties of a film for scientific study

is not unrelated to the optimization of the material for use as a conductor in electromagnets, and the solution to the problem of preparing tunneling junctions is certainly related to the problem of preparing practical devices.

II. PROGRESS

A. Facilities

The major effort related to facilities has been the upgrading of the multi-source electron beam deposition system which is used to prepare the ternary and pseudoternary compounds that are being studied under the program. Specificially, a sample insertion system has been designed, fabricated, and tested successfully. The vacuum lock of this system is presently pumped by a 100 l/s ion pump, which will be replaced in the near future by a turbomolecular pump. The latter should permit even more rapid turn-around than currently possible.

The vacuum chamber has been equipped with a Williamson[4] noncontacting temperature sensing system for monitoring substrate temperature. With this system the temperature of the substrate is determined by computing the ratio of radiant energies emitted from the surface in two adjacent wavebands. In this way the results are independent of the emissivity of the substrate. The signals are processed through one silicon detector for long-term stability and drift-free operation. The electronics are outside the vacuum system with the viewing of the sample surface accomplished with a fiber optic link. With this instrument it should be possible to greatly tighten the reproducibility of the control of substrate temperature which appears to be a critical factor in the control of the properties of films.

We have not purchased a rate monitor which uses Electron Impact Emission Spectroscopy (EIES). The currently commercially available instrument, supplied by Inficon, has the capability for controlling only two sources, whereas in our applications there is a need for control of up to four. We have discovered that the limitation of our currently-used crystal oscillator rate/thickness monitoring system, saturation of the crystal which monitors the sulfur flux, can be overcome by periodically heating the crystal to evaporate the sulfur which may have collected on it. We have thus decided to continue to use collimated crystal oscillator rate/thickness monitors. We will replace the DCC-116 minicomputer used to monitor deposition rates with a suitably equipped microcomputer such as an IBM PC. This will be done to avoid delays and possibly large expenses that might be associated with the maintenance of the DCC microcomputer which is a copy of a Data General NOVA 1200. This change in computers will be made before 1 July 1985. An account of the operation of the deposition system has been submitted for publication in the Journal of Vacuum Science and Technology.

B. Work on the Chevrel Phase Compounds

The experimental effort in this area has been concentrated on the study of $HoMo_6S_8$ thin films prepared during the fall. $HoMo_6S_8$ is a reentrant superconductor[6] and is an excellent model system for the study of the interplay of ferromagnetism and superconductivity. The studies of these films, which will be described below, are preliminary to electron tunneling investigations.

A technique which we have called reactive annealing has been developed.[7] After a film has been made and characterized it is possible to

reinsert it into the vacuum system, and in the the presence of a flux of sulfur vapor anneal it. This process can be repeated. It was found that as-prepared $HoMo_6S_8$ films were barely metallic and did not undergo a complete transition into the superconducting state. Annealing for an hour at 800°C resulted in reentrant superconductivity very similar to that observed in bulk polycrystalline samples. Further annealing resulted in a further reduction of the resistivity of the film, an increase in its resistivity ratio and the disappearance of the reentrance into the normal state below 1 K.

Extensive measurements of the resistance as a function of temperature and magnetic field have been carried out to determine the nature of the superconducting state at low temperatures in non-reentrant films. It is found that in these films reentrance into the normal state actually occurs in a field of 200 G. At temperatures of the order of 0.1 K the time constants of the films in response to changes in the magnetic field appear to be the order of one hour. This is the case for films which are only 1/2 μm thick.

At the present time we believe that the remarkable low temperature behavior of these films has two possible causes. Zero resistance may be a consequence of domain boundary superconductivity first discussed by Tournier and co-workers.[8] Alternatively it may result from the transformation of $HoMo_6S_8$ from a ferromagnet at low temperatures to an antiferromagnet possibly as a consequence of the depletion of Ho in the annealing process.[9] The latter will be checked shortly by chemical analysis of the samples. By studying the critical fields of the films in both parallel and perpendicular fields it should be possible to infer the temperature dependence of the magnetization.[10] If this can be done with sufficient precision, the nature of the low

temperature magnetic phase which appears to coexist with superconductivity in this peculiar manner will be ascertained.

A preliminary account of this work will be presented at the March meeting of the American Physical Society. The work will also be presented at the Conference on Superconducting Mechanisms and Structures which will be held in Ames, Iowa in the late spring of 1985. It is hoped that by the time of this second meeting a definitive explanation of these unusual results will be in hand.

C. Heavy Fermion Superconductors.

These materials have attracted attention because of the remarkable fact that the effective masses of the electron quasiparticles can be as much as a thousand times larger than the free electron effective mass. An important consequence of this is that the Debye temperature of some of these materials can be larger than or comparable to the Fermi temperature. Thus many of the steps used in simplifying the theoretical analyses of solids are no longer valid. In the case of heavy fermion materials which are superconducting it is highly probable that the usual mechanism for superconductivity involving a retarded effective electron-electron interaction moderated by the phonons is not valid. Some investigators have suggested that heavy fermion superconductors may involve higher spin and angular momentum pairing than is the case for usual BCS superconductors.[11]

We have undertaken the study of the superconductivity of UPt_3 thin films. This compound is a heavy fermion superconductor which may actually be a triplet superconductor. Our efforts have been concentrated on preparing thin films using a sputtering technique which could be suitable for electron

tunneling investigations. The latter, when incorporated as electrodes in tunneling junctions, could be used to measure the energy gap of this remarkable material. If the junctions were of sufficiently high quality, the coupling between the electrons and the magnetic and vibrational internal degrees of freedom of the compound could be investigated in a quantitative way. If Josephson junctions could be made, it might be possible to learn something about the details of the pairing.

We have solved one major technical problem in the preparation of UPt films, and are trying to solve a second. In all of our early efforts at preparing UPt_3 we always obtained oxides of uranium rather than the desired compound. We discovered that uranium was reacting with the oxygen in the sapphire substrates at the high temperatures needed to form UPt_3. We were able to form UPt_3 on tungsten substrates, on tungsten-coated sapphire substrates, and on Berylia (BeO) substrates. However, although the X-ray patterns of these films were reasonably sharp, their resistivity ratios were not high enough to result in superconductivity at any attainable temperature. Attempts at annealing were not successful for a variety of reasons. In the case of the Berylia and tungsten substrates the disordered film doesn't anneal because it is pinned by the roughness of the substrate, whereas in the case of tungsten-coated sapphire substrates, tungsten appears not to be an adequate barrier against the interaction of the UPt_3 film with the oxygen in the sapphire substrate. What appears to be necessary for successful annealing is a smooth, single-crystal substrate which doesn't react with uranium. The success of this enterprise depends on finding such a material and successfully preparing either a single crystal films or films with large enough crystallites that their resistivity ratios are high.

Scrutiny of standard tables of free energies of formation of binary compounds reveals why BeO and W are suitable substrates for the reactive deposition of UPt_3 at relatively high temperatures.[12] Using the tables, we have also identified CaF_2 as a suitable substrate material. It will not react with uranium under the deposition conditions needed to form UPt_3 and is available in the form of polished single crystals.[13] In the near future we will be sputter-depositing UPt_3 films onto this material. If the process is successful, we will be in an excellent position to carry out quantitative microscopic studies of the mechanisms of superconductivity in UPt_3.

D. Low-Carrier Density Superconductors and the Field Effect

The demise of the superconducting computer project at IBM was in part a consequence of a three-terminal superconducting switching device with FET-like characteristics not having been developed. Josephson devices require design rules entirely different from those of semiconductor devices and were two-way devices. In addition, the circuit concepts requied to apply the Josephson effect, because they involved magnetic-field driven switching, were not easily incorporated into high-density systems. If a three-terminal superconducting switch with isolation, high-speed, low-power dissipation and gain could be developed, then the perceived advantages of superconducting digital electronics relating to lower power dissipation, high packing density and ease of interconnection might be practically realized.[14]

It may be possible to produce a superconducting device with FET-like characteristics using the field effect in low carrier density superconductors. In order for the effect to be significant, a low carrier concentration system is needed because the field effect in metals is extremely

weak. $BaPb_{1-x}Bi_xO_3$ is a prototype low carrier density material with a superconducting transition temperature above 10 K.[15] However, problems in fabricating it in a controlled way seem to preclude its use in a simple demonstration of the field effect. On the other hand, PbTe doped with Tl is a relatively simpler material with a transition temperature above 1.4 K which may be very useful in demonstrating the field effect even if it is not actually useful in practical devices.[16]

If the areal carrier density of a film is sufficiently low, then it should be possible to effect a large percentage change in its surface charge density using the field effect. In a material such as PbTe doped with Tl, in which T_c = 1.5 K and the carrier concentration is 10^{19} cm^{-3}, large changes in the areal charge carrier density can be induced with a bias of the order of a volt. It may thus be possible to switch superconductivity on and off in a film by biasing it with a voltage applied across an insulating layer using a gate electrode.

We have undertaken a design study and a pilot project involving PbTe to see whether it is possible to produce a significant change in the superconductivity using an applied potential. Subsequent to our work on this project we discovered an analysis by Shapiro of surface superconductivity induced by an electric field.[17] Although this analysis was focussed on a low transition temperature material $SrTiO_3$, its results were essentially identical to our own. We plan to produce Tl-doped PbTe films by a sputtering process and have been preparing masks, substrates and sputtering targets. We anticipate some tangible experimental results within the next few months. A brief outline of the idea is attached as Appendix A.

III. TECHNICAL FORECAST

A. Chevrel Phase Compounds (Maps, Berkely, Kang, Goldman)

We will continue our studies of $HoMo_6S_8$, expanding the work to include tunneling investigations of this compound. Since there are no quantitative studies of tunneling in any magnetic superconductor, this sort of investigation should be of some significance.

We plan to extend and substantially broaden the scope of our investigation of other Chevrel phase films, taking advantage of our unique sample preparation facilities. We will prepare additional $CuMo_6S_8$ films and attempt to carry out quantitative studies of electron tunneling. $CuMo_6S_8$ is a high-critical field material. We will also prepare the pseudo-ternary compound, europium-tin-molybdenum sulfide, and study superconductivity induced by high external magnetic fields.[18] This phenomenon is supposed to involve spin compensation resulting from a negative exchange interaction between the conduction electrons and magnetic ions. The effect, originally proposed by Jaccarino and Peter,[19] may be a key to achieving superconductivity in ultra-high magnetic fields. We will study the systematics of the phenomenon as a function of temperature and magnetic field as well as carry out tunneling studies in a magnetic field.

B. Heavy Fermion Superconductors (Kang, Maps, Goldman)

If the fabrication procedure involving the use of polished single crystal substrates of CaF_2 results in films with high resistivity ratios, then this project will proceed to the stage of characterization of the macroscopic superconducting properties of the films and attempts to fabricate high-quality

tunneling junctions will be initiated. If films of the appropriate quality cannot be prepared, the effort will be dropped and the resources will be channeled into other aspects of the program. This effort, if successful, could result in a major contribution to the fundamental understanding of heavy fermion superconductors.

C. Low Carrier Density Superconductors and the Field Effects (Jaeger, Goldman)

We plan to fabricate films of PbTe doped with Tl with carrier concentrations of the order of 10^{8} to 10^{19} cm^{-3}. This will be done using a sputtering technique. With a suitable electrode configuration we should be able to bring about a shift of T_c resulting from the change of the carrier concentration due to the field effect. This effort should be completed some time during the present grant period. If this pilot effort is sufficiently successful, we will expand the effort in this area as the development of a three-terminal FET-like superconducting device would represent a major breakthrough in superconductive electronics. This expansion could involve the preparation of films and devices using MBE techniques. The use of these techniques should lead to higher quality films and devices, and perhaps to higher T_c's for the low carrier density material.

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IV. PERSONNEL

A. A. M. Goldman, Professor of Physics

B. J. Maps, Research Associate in Physics

C. J. Kang, Research Assistant in Physics

D. D. Berkely, Research Assistant in Physics

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Appendix A

A Three-Terminal Superconducting Field Effect Switch (SuperFET)

B. G. Orr, H. M. Jaeger, and A. M. Goldman
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A major problem in the use of superconducting digital technology is the lack of a high-speed, low-power three terminal device with properties similar to those of a semiconducting field effect transistor (FET). Specifically such a device has three-terminals, a high input impedance, input-output isolation, low power dissipation, and a very fast switching speed. Josephson devices have been used as building blocks of digital superconducting circuitry, but they require unusual design rules and there have been problems in achieving fabrication tolerances on various parameters which are consistent with the margins required by circuit design considerations. Many important Josephson digital circuit configurations, especially those used in memory applications, are based on the SQUID configuration which cannot be easily miniaturized because if they were the magnetic field associated with the flux quantum would become prohibitively large.

Various three-terminal superconducting devices are discussed in the attached reprints. The so-called superconducting FET is a device which is just like an ordinary FET, but relies on the superconducting proximity effect to

induce superconductivity in the conducting channel. Because of the low carrier density in the channel, the superconducting source and drain electrodes must be very close to each other in order to achieve any effect at all. Here we propose a totally different approach to the problem of constructing a superconducting FET. There is a small class of doped semiconductors or semimetals which, although having low charge carrier concentrations, are nevertheless superconductors. The most well-known and intensively studied is $PbBa_xB_{1-x}O_3$, which can have a superconducting transition temperature up to 13 K, depending upon the carrier concentration which is ordinarily set by x. Remarkably these materials, even with $T_c \sim 13$ K, have carrier concentrations n ≃ $\simeq 10^{19}$ cm^{-3}. There are also a number of semiconductors with very low carrier concentrations which can be doped so that they are superconductors. With the exception of PbTe doped with Te, all of these materials have transition temperatures less than 1 K. This compound has a T_c which can be as high as 2 K.

An important feature of all of these low carrier density superconductors is that T_c is changed (increased) as the carrier concentration is changed (increased) by doping or altering the chemical composition. An alternative way to vary the carrier concentration, at least at a surface layer, is to use the field effect. The material of interest is incorporated as one electrode of a capacitor which, when biased, results in negative or positive charges being stored. For a metal these charges would be confined to a surface layer. Also the charge density change resulting from biasing up to the breakdown electric field of the dielectric at most would produce a change in the charge density of a metal by at most a part in 10^4. Consequently, the metal field effect, which is the phenomenon actually involved in this instance, will have

a barely perceptible affect on the superconducting transition temperature. The possibility of changing T_c by electrically charging a superconducting film was demonstrated by Glover a number of years ago. In this instance the magnitude of the change in T_c resulting from biasing the material would be expected to be orders of magnitude larger than what was observed by Glover.

The proposed device would be a thin film of a semiconducting semimetal which is a superconductor.

Our pilot studies will involve the use of PbTe doped to give $T_c \sim 1.5$ K. There would be two superconducting electrodes attached to the film. This structure would be covered with an insulating layer about 100 Å thick. A third superconducting electrode would be deposited over the insulator to form a capacitor with the underlying PbTe film. Bias would be applied between the PbTe layer and this upper film. The operation of the device would involve stabilizing its temperature above T_c of PbTe and then inducing superconductivity in the latter with the application of a voltage bias to the capacitor plates. The device could operate by setting the temperature below T_c and using a reverse bias, to deplete the carrier concentration in the PbTe film thus shutting off the superconductivity. Either scheme would result in the realization of a switch, between the normal and superconducting states of the PbTe or other suitable material, where the control gate had a very high impedance, limited by leakage and tunneling through the dielectric layer. The time constant would be limited by the RC product of the gate where R is the resistance of the input circuitry and C is the capacitance of the control gate with respect to the PbTe layer. This time would be as short as that achieved in any FET type of device fabricated using Si or GaAs technology.

The power dissipated in the device would be the I^2R product in the normal state of the PbTe conducting channel. This could be optimized by a suitable choice of biasing conditions. There would be no power dissipated when the PbTe film was in the superconducting state.

It is useful to estimate the magnitude of the potential change in the carrier concentration of the PbTe layer as a result of the field effect. We assume a 100 Å thick PbTe film separated from the control electrode by a 100 Å thick insulating layer. We also assume that the PbTe film has a T_c of 1.5 K and a carrier concentration of 10^{19} cm^{-3}. We assume a 1 volt bias. Then the total charge Q transferred is given by

$$Q = CV \tag{1}$$

where C is the capacitance which is

$$C = \frac{A\varepsilon}{d} = V \tag{2}$$

Then Eq. (1) can be rewritten as

$$N = \frac{Q}{Ae} = \frac{\varepsilon}{e} = \frac{V}{d} \tag{3}$$

where ε is the dielectric constant of the insulator, $\frac{V}{d}$ is the electric field, e is the charge on an electron and N is the carrier concentration/area. Taking $\varepsilon = 10\ \varepsilon_o$, a reasonable value for a dielectric such as Al_2O_3 or SiO and $\varepsilon_o = \frac{1}{36}\pi \times 10^{-9}$ in SI units we find $N = 5.5 \times 10^{14}/cm^2$. The areal charge density in a 100 Å thick PbTe film with $n = 10^{19}/cm^3$ is 10^{13} cm^2. Thus the bias will increase the carrier concentration in the inversion layer of PbTe by a factor of 50 over its original value. This <u>may</u> be sufficient to bring about superconductivity in the film or in a significant fraction of its cross section. The uncertainty is in the extent to which the charge added to the PbTe electrode penetrates away from the surface. The more insulating the character of the nmormal state behavior, the deeper the penetration and the

greater the likelihood of success. A possibility is to employ a thinner PbTe film, but such a choice may compromise the power dissipation in the normal state.

The demonstration of feasibility will consist of demonstrating that the superconducting state in PbTe can be induced by the application of a bias voltage to the structure described above. If this can be done, then serious studies relating to finding better materials and optimizing the geometry become relevant.